Standard Operating Procedure for GLNPO Total Alkalinity Titration

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1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters; domestic and industrial wastes.

1.2 This method is configured for water in the range of 10 to 250 mL/L total alkalinity as CaCO3.

2.0 Summary of Method

This procedure for total alkalinity is an adaptation of the technique outlined in Standard Methods for the Analysis of Water and Waste Water. A measured amount of sample is titrated with acid to a pH of 4.5.

3.0 Sample Handling and Preservation

Glass or plastic containers are suitable. A representative unaltered aliquot is used. Biological activity could modify the nitrogen balance and therefore slightly alter the total alkalinity, if the sample is not analyzed immediately.

4.0 Interferences

4.1 Oil and grease may interfere by coating the electrodes and causing a sluggish response.

4.2 High mineral content may interfere by altering the activity of the water.

5.0 Apparatus

5.1 pH meter with a combination electrode.

5.2 Buret 25 mL auto zero.

5.3 Variable speed stirring motor and glass stirring paddle. The speed of this stirring apparatus should be adjusted each time it is used such that the solution is stirring rapidly, but not so rapid that the surface is broken.

5.4 Beaker 150-200 mL.

6.0 Reagents

6.1 Standard titrant - 0.0200 N H2SO4 (commercial). Alternatively, a more concentrated commercial standard(1.0 N or 0.8 N) can be diluted volumetrically on site to 0.0200 N with reagent water.

6.2 Standard pH buffers 4.0 and 7.0. Prepare from concentrates or powders as described with the product. Use graduated cylinders for dilutions.

6.3 Stock alkalinity control standard: Dissolve 4.24 gm of Na2CO3 (dried at 250°C for two hours and cooled in a desiccator) in reagent water and dilute to one liter in a volumetric flask. Use this solution to prepare control standards by dilution with volumetric labware.

6.4 Typical control standards for the working range of 10-250 mg alkalinity per liter may be prepared as follows.

mL stock diluted to 1 L	Concentration mg alk/per liter as CaCO3
20	80
25	100

7.0 Procedure

7.1 pH meter calibration.

7.1.1 Bring ph buffers 7.0 and 4.0 to 25°C ± 5°C. Set the temperature control knob to 25°C.

7.1.2 With pH 7.0 buffer on electrode and stirrer on, adjust calib. control so meter reads 7.0.

7.1.3 With pH 4.0 buffer on electrode and stirrer on, adjust amplification or gain on meter so that meter reads 4.0.

7.1.4 Repeat Steps 7.1.2 through 7.1.3 above until no further adjustment is necessary.

7.2 pH meter daily check.

7.2.1 With pH 4.0 buffer on the electrode, and stirrer on, adjust the calib knob so meter reads 4.0.

7.2.2 With pH 7.0 buffer on the electrode and the stirrer on, check to be sure that the meter reads 7.0. If not then perform the pH meter calibration procedure above.

7.3 Titrate 100 mL of sample or check standard (modified 100 mL volumetric flask) with the 0.0200 N H2SO4 to pH 4.5 using moderately vigorous stirring action near the end of the titration. The stirring action should be vigorous enough near the end of titration to break the surface to allow rapid equilibrium of CO2 between the solution and the atmosphere.

8.0 Calculations

Total Alkalinity as CaCO3 in mg/L= (ml of titrant) X 10

9.0 Quality Control

9.1 GLNPO Total Alkalinity

The two control standards described above are run once per 12 hour shift, or once every two stations, whichever is less. Reagent blanks (reagent water processed through the sample storage container) are run approximately once in every four stations.

10.0 Preventative Maintenance

10.1 This is described in the laboratory logbook.

10.2 Maintain pH 7.0 buffer on the electrode when not in use.

11.0 References

11.1 EPA Publication, March 1979. "Methods for Chemical Analysis of Water and Wastes". EPA #600/4-79-02.

11.2 Standard Methods for the Analysis of Water and WasteWater, 16th Edition APHA-AWWA-WPCF.